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WADC TECHNICAL REPORT 54-46

AN INVESTIGATION OF
LITHIUM 9/10 HYDROXYSTEARATE GREASES

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MATERIALS LABORATORY

AUGUST 1954

WRIGHT AIR DEVELOPMENT CENTER

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Materials Laboratory

August 1954

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Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared by the Petroleum Products Branch and was initiated under Research and Development Order No. 613-11(E-A), "Aircraft Lubricating Greases". The report was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Lt H. C. Markle and Mr. H. Schwenker acting as project engineer.

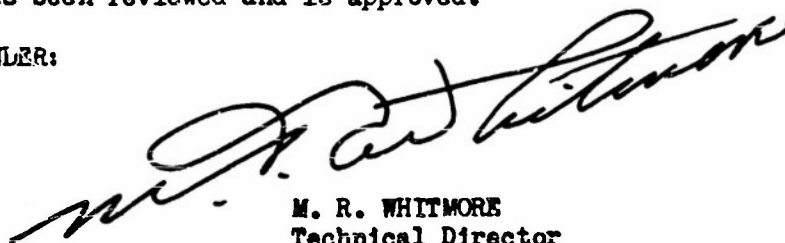
ABSTRACT

The melting points of lithium soaps, which were prepared from hydroxystearic acids, were determined. These soaps were combined with synthetic oils to prepare greases which, in turn, were analyzed to determine their physical and chemical properties. Except for water resistance, oil separation, and mechanical stability, the greases made from lithium 9 or 10 hydroxystearate were found to possess properties equal to those of lithium 12 hydroxystearate.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

TABLE OF CONTENTS

	Page
SUMMARY	1
SECTION I - INTRODUCTION	1
SECTION II - PREPARATION OF LITHIUM HYDROXYSTEARATE SOAPS	1
SECTION III - PREPARATION OF LITHIUM HYDROXYSTEARATE GREASES	2
SECTION IV - PHYSICAL AND CHEMICAL EVALUATIONS OF LITHIUM HYDROXYSTEARATE GREASES	4
Mechanical Stability	4
Oil Separation	15
Water Resistance	16
Wear	16
Oxidation Stability	17
Copper Corrosion	18
Melting Points	18
SECTION V - IMPROVEMENT OF MECHANICAL STABILITY	19

LIST OF TABLES

	Page
TABLE I - GREASE FORMULATION	3
TABLE II - MECHANICAL STABILITY OF GREASES	4
TABLE III - OIL SEPARATION OF GREASES	15
TABLE IV - WATER RESISTANCE OF GREASES	16
TABLE V - WEARING CHARACTERISTICS OF GREASES	17
TABLE VI - OXIDATION STABILITY OF GREASES	17
TABLE VII - MELTING POINTS OF GREASES	18
TABLE VIII - MECHANICAL STABILITY OF IMPROVED GREASES	20

LIST OF ILLUSTRATIONS

	Page
Figure 1. Unworked Sample of MLG-6833 (12% Lithium 12 Hydroxystearate and 88% Tri decyl azelate)	6
Figure 2. Worked Sample (500 Strokes by Hain Microworker) of MLG-6833 (12% Lithium 12 Hydroxystearate and 88% Tri decyl azelate)	7
Figure 3. Unworked Sample of MLG-6834 (12% Lithium 12 Hydroxystearate and 88% Dipropylene glycol dipelargonate)	8
Figure 4. Worked Sample (500 Strokes by Hain Microworker) of MLG-6834 (12% Lithium 12 Hydroxystearate and 88% Dipropylene glycol dipelargonate)	9
Figure 5. Unworked Sample of MLG-6812 (12% Lithium 12 Hydroxystearate and 88% Di (2 ethyl hexyl) sebacate)	10
Figure 6. Unworked Sample of MLG-6836 (12% Lithium 9/10 Hydroxystearate and 88% Di (2 ethyl hexyl) sebacate)	11
Figure 7. Worked Sample (500 Strokes by Hain Microworker) of MLG-6836 (12% Lithium 9/10 Hydroxystearate and 88% Di (2 ethyl hexyl) sebacate)	12
Figure 8. Unworked Sample of MLG-6838 (12% Lithium 9/10 Hydroxystearate and 88% Di (2 ethyl hexyl) sebacate)	13
Figure 9. Worked Sample (500 Strokes by Hain Microworker) of MLG-6838 (12% Lithium 9/10 Hydroxystearate and 88% Di (2 ethyl hexyl) sebacate)	14
Figure 10. Unworked Sample of MLG-9771 (15% Lithium 9/10 Hydroxystearate and 85% Di (2 ethyl hexyl) sebacate)	21
Figure 11. Worked Sample (500 Strokes by Hain Microworker) of MLG-9771 (15% Lithium 9/10 Hydroxystearate and 85% Di (2 ethyl hexyl) sebacate)	22
Figure 12. Unworked Sample of MLG-9801 (3% Lithium 12 Hydroxystearate, 12% Lithium 9/10 Hydroxystearate, and 85% Di (2 ethyl hexyl) sebacate)	23

SUMMARY

Samples of 9/10 hydroxystearic acid were submitted by the United States Department of Agriculture and Emery Industries, Inc. to the Materials Laboratory for evaluation as grease-thickening agents. Only slight differences exist between the properties of these acids and the lithium soaps made from them. Melting points of the lithium 9/10 hydroxystearate soaps compare favorably with lithium 12 hydroxystearate. The soaps of these acids produce a somewhat softer grease than that produced by the same percentage of 12 hydroxystearic acid soap when used with the same base-oil; but they appear to be good gelling agents.

Preliminary investigations revealed that lithium 9/10 hydroxystearate greases compared favorably with lithium 12 hydroxystearate greases except for mechanical stability, oil separation, and water resistance. These deficiencies probably can be corrected by further study.

Initial attempts to improve the mechanical stability of lithium 9 or 10 hydroxystearate greases have been encouraging; consequently, future work will be devoted to study along this line in order to formulate greases that will meet the specification requirements of current aircraft greases.

SECTION I

INTRODUCTION

Many greases currently used in the Air Force are made from materials that are imported or whose supply is limited. In the event of war the source of supply may be cut off or become insufficient for the needs of the Air Force. It is desirable, therefore, to use non-critical materials which are obtained from domestic sources in the development of greases which are as good or better than present Air Force lubricating greases. This task is to be accomplished by the investigation of 9/10 hydroxystearate soaps, non-soap thickeners, and synthetic oils other than those derived from castor oil.

The materials evaluated as grease-thickening agents in this investigation were the lithium soaps of 12 hydroxystearic acid and 9/10 hydroxystearic acid. Some aircraft lubricating greases in current use employ 12 hydroxystearic acid soaps as thickening agents. These acids are derived from castor oil, an imported and expensive material. Tallow, an inexpensive domestic product, is the raw material from which 9/10 hydroxystearic acid is obtained.

SECTION II

PREPARATION OF LITHIUM HYDROXYSTEARATE SOAPS

Lithium soaps were prepared from the following acids:

- (1) 9/10 hydroxystearic acid (MLG-6443); melting point, 66.5° to 73.5°C; supplied by Emery Ind., Inc.

- (2) 9/10 hydroxystearic acid (MLG-6395); melting point, 70.0° to 75.0°C; supplied by NRRL Dept. of Agric.
- (3) 12 hydroxystearic acid (MLG-6855); melting point, 75.0° to 76.0°C; supplied by Emery Ind., Inc.

A lithium hydroxide base, manufactured by Metalloy Corp., was also used in the preparation of the soap; but although labeled anhydrous, it proved to be an old sample and was found to contain a small percentage of water.

A predetermined amount (usually 100 gm) of the acid was placed in a one-liter, stainless steel beaker. Distilled water was added to the acid until the beaker was about half full. Then, the beaker was heated in an oil bath until the mixture was slightly warmer than the melting point of the acid. The mixture was stirred by an air-driven, glass stirrer.

An equivalent weight of lithium hydroxide, having been previously dried (or corrected for a known moisture content), was placed in a separate beaker. Distilled water was added; the solution was warmed and then decanted into the acid mixture in the stainless steel beaker. This process of adding water and decanting the solution was repeated until all the base was added to the acid mixture.

After heating (80° — 90°C) and stirring for at least one hour, this mixture was allowed to cool; it was then filtered through a Buchner funnel. The soap that collected in the filter was returned to the stainless steel beaker; more distilled water was added; and the mixture was heated and stirred for at least another hour. This mixture was allowed to cool and was then refiltered through a Buchner funnel.

The soap was dried in an oven at 160° to 210°F for at least 24 hours.

The following melting points were observed for the lithium soaps supplied by Emery Ind., Inc., NRRL Dept. of Agric., and Emery Ind., Inc., respectively:

- (1) lithium 9/10 hydroxystearate (MLG-6859) 201° — 204.5°C
- (2) lithium 9/10 hydroxystearate (MLG-6858) 207° — 210°C
- (3) lithium 12 hydroxystearate (MLG-6857) 207° — 208.5°C

SECTION III

PREPARATION OF LITHIUM HYDROXYSTEARATE GREASES

Greases were made from MLG-6859, MLG-6858, MLG-6857, and the following oils: di-(2 ethyl hexyl) sebacate (Distilled), supplied by Rohm and Haas Co.; tri decyl azelate [bis(tridecyl) azelate], supplied by Emery Ind., Inc.; dipropylene glycol dipelargonate, supplied by Emery Ind., Inc.

A predetermined amount (18 gm) of the lithium-base soap was weighed in a stainless steel beaker to which the proper amount of oil (132 gm) was then added to give the desired percentages of soap and oil. The beaker containing the mixture was then preheated on a hot-plate to about 170°C, and then placed in an oil bath which was held at 220° to 230°C. The soap and oil mixture was stirred until the soap had melted completely into the oil (190° to 200°C.). The beaker was removed from the oil.

bath; and the mixture was quickly quenched by pouring it on a stainless steel table top. The resulting grease was milled three times through a three-roll paint mill having a clearance between rolls of about 0.002 inches.

Table I shows the different greases that were made by combining the thickeners (lithium soaps) with the oils in the proportion of 12 and 88%, respectively. Duplicate batches of each grease were made to check their reproducibility.

TABLE I
GREASE FORMULATION

IDENT NO.	LITHIUM SOAP	OIL	MICRO PENETRATION
MLG-6701	MLG-6857	di(2 ethyl hexyl)sebacate	62
MLG-6702	MLG-6859	di(2 ethyl hexyl)sebacate	135
MLG-6836	MLG-6859	di(2 ethyl hexyl)sebacate	164
MLG-6703	MLG-6858	di(2 ethyl hexyl)sebacate	122
MLG-6838	MLG-6858	di(2 ethyl hexyl)sebacate	81
MLG-6704	MLG-6858	dipropylene glycol dipelargonate	65
MLG-6837	MLG-6858	dipropylene glycol dipelargonate	147
MLG-6705	MLG-6859	dipropylene glycol dipelargonate	72
MLG-6835	MLG-6859	dipropylene glycol dipelargonate	95
MLG-6714	MLG-6857	dipropylene glycol dipelargonate	45
MLG-6834	MLG-6857	dipropylene glycol dipelargonate	43
MLG-6753	MLG-6858	tri decyl azelate	70
MLG-6778	MLG-6858	tri decyl azelate	66
MLG-6754	MLG-6859	tri decyl azelate	81
MLG-6773	MLG-6859	tri decyl azelate	72
MLG-6755	MLG-6857	tri decyl azelate	42
MLG-6833	MLG-6857	tri decyl azelate	45

The gelling power of the lithium 9/10 hydroxystearates proved to be less than that of lithium 12 hydroxystearate. With a few exceptions, reproducibility of the greases (determined by their initial consistency) was good. For those cases in which the consistency varied for different batches of the same material, the greases were made from lithium 9/10 hydroxystearate and di(2 ethyl hexyl)sebacate or lithium 9/10

hydroxystearate and dipropylene glycol dipelargonate. This was probably due to a false consistency which is sometimes obtained with a low percentage of soap when using this type of quenching, that is, when chilling rapidly on a stainless steel sheet. It will be noted that where the tri decyl azelate was used with the lithium 9/10 hydroxystearate the reproducibility of different batches of this grease was good. This can be attributed to the higher viscosity of tri decyl azelate which made the percentage of soap used in this case adequate.

SECTION IV

PHYSICAL AND CHEMICAL EVALUATION OF LITHIUM HYDROXYSTEARATE GREASES

Mechanical Stability

The mechanical stability of the greases was checked in the Hain Microworker. Lithium 12 hydroxystearate exhibited excellent mechanical stability, whereas the mechanical stability of the lithium 9/10 hydroxystearate was only fair.

Electron micrographs were made of the greases to determine if the better mechanical stability of the lithium 12 hydroxystearate grease was due to a difference in fiber structure. Examination of the electron micrographs revealed that the lithium 12 hydroxystearate (Figures 1 through 5) had a twisted, symmetrical fiber structure which has been credited with giving good mechanical stability. The lithium 9/10 hydroxystearate (Figures 6 through 9) had a rod-like structure, and the fibers were packed together in bundles. After working, both the lithium 12 hydroxystearate and the lithium 9/10 hydroxystearate fibers appear to be grouped closer together.

The lithium 12 hydroxystearate fibers, when packed together, do not form a solid mass, but give a lattice-type structure with many interstices which present a large surface area of the thickener for retention of the oil; this results in a very slight breakdown of the grease due to working.

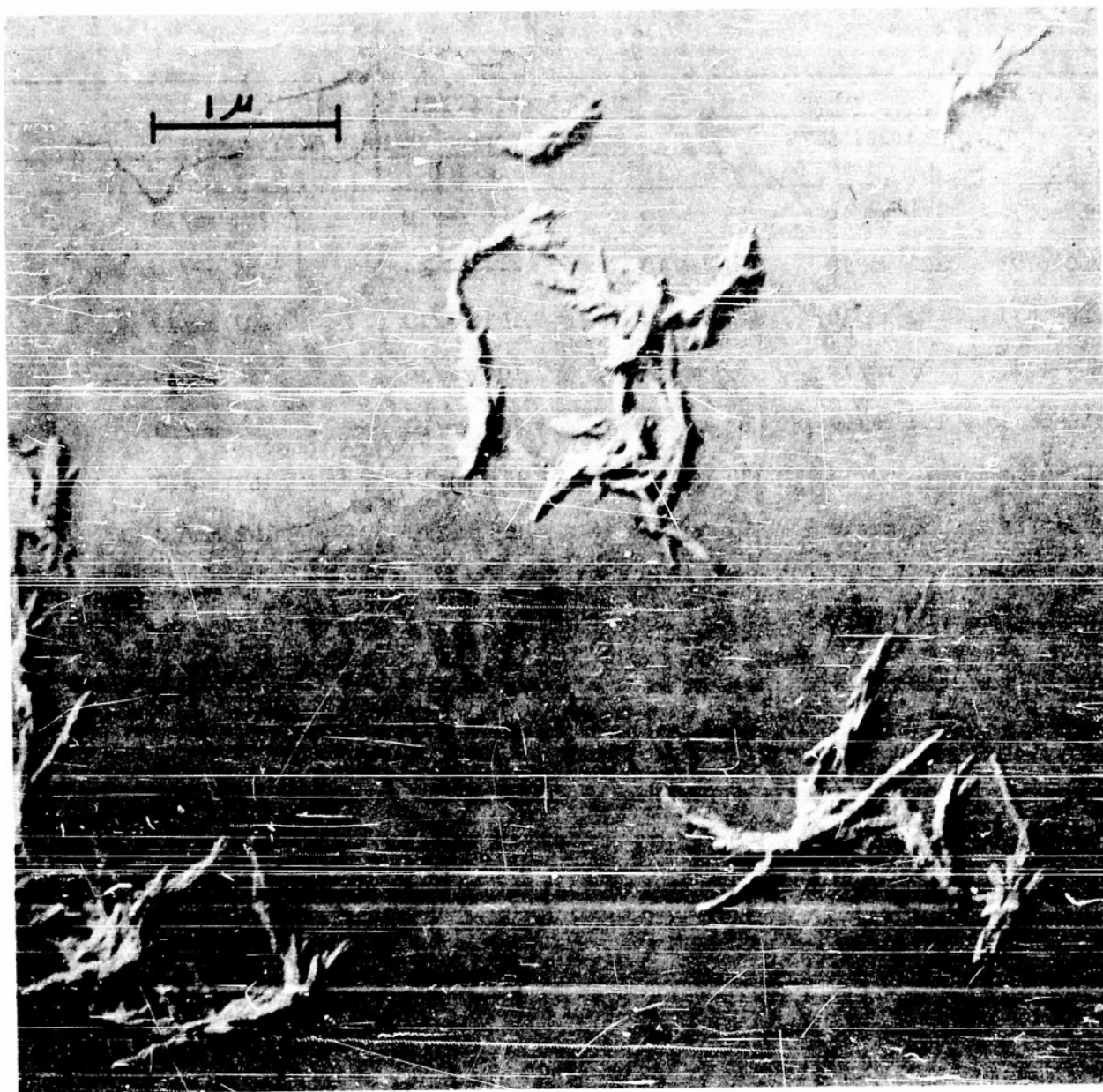
The fibers of lithium 9/10 hydroxystearate are also packed together on working, but, here, a more solid mass is formed, reducing the surface area of the thickener which results in a reduction of its oil retaining ability. This causes a greater breakdown of lithium 9/10 hydroxystearate greases when worked. The micropenetrations of the lithium hydroxystearate greases are listed in Table 2.

TABLE II
MECHANICAL STABILITY OF GREASES

IDENT NO.	MICROPENETRATION			
	Initial ASTM	Strokes Worked in Hain Microworker		
		50	100	500
MLG-6701	62 mm/10	74 mm/10	71 mm/10	94 mm/10
MLG-6702	135 mm/10	172 mm/10	—	206 mm/10

TABLE II (Continued)

IDENT NO.	MICROPENETRATION			
	Initial ASTM	Strokes Worked in Hain Microworker		
		50	100	500
MLG-6836	164 mm/10	257 mm/10	248 mm/10	260 mm/10
MLG-6703	122 mm/10	151 mm/10	————	185 mm/10
MLG-6838	81 mm/10	129 mm/10	143 mm/10	207 mm/10
MLG-6704	65 mm/10	89 mm/10	108 mm/10	130 mm/10
MLG-6837	147 mm/10	253 mm/10	266 mm/10	304 mm/10
MLG-6753	70 mm/10	115 mm/10	115 mm/10	143 mm/10
MLG-6778	66 mm/10	101 mm/10	115 mm/10	142 mm/10
MLG-6705	72 mm/10	106 mm/10	146 mm/10	153 mm/10
MLG-6835	95 mm/10	145 mm/10	157 mm/10	195 mm/10
MLG-6754	81 mm/10	128 mm/10	142 mm/10	164 mm/10
MLG-6773	72 mm/10	100 mm/10	122 mm/10	140 mm/10
MLG-6714	45 mm/10	56 mm/10	59 mm/10	72 mm/10
MLG-6834	43 mm/10	50 mm/10	54 mm/10	58 mm/10
MLG-6755	42 mm/10	52 mm/10	57 mm/10	69 mm/10
MLG-6833	45 mm/10	55 mm/10	43 mm/10	65 mm/10



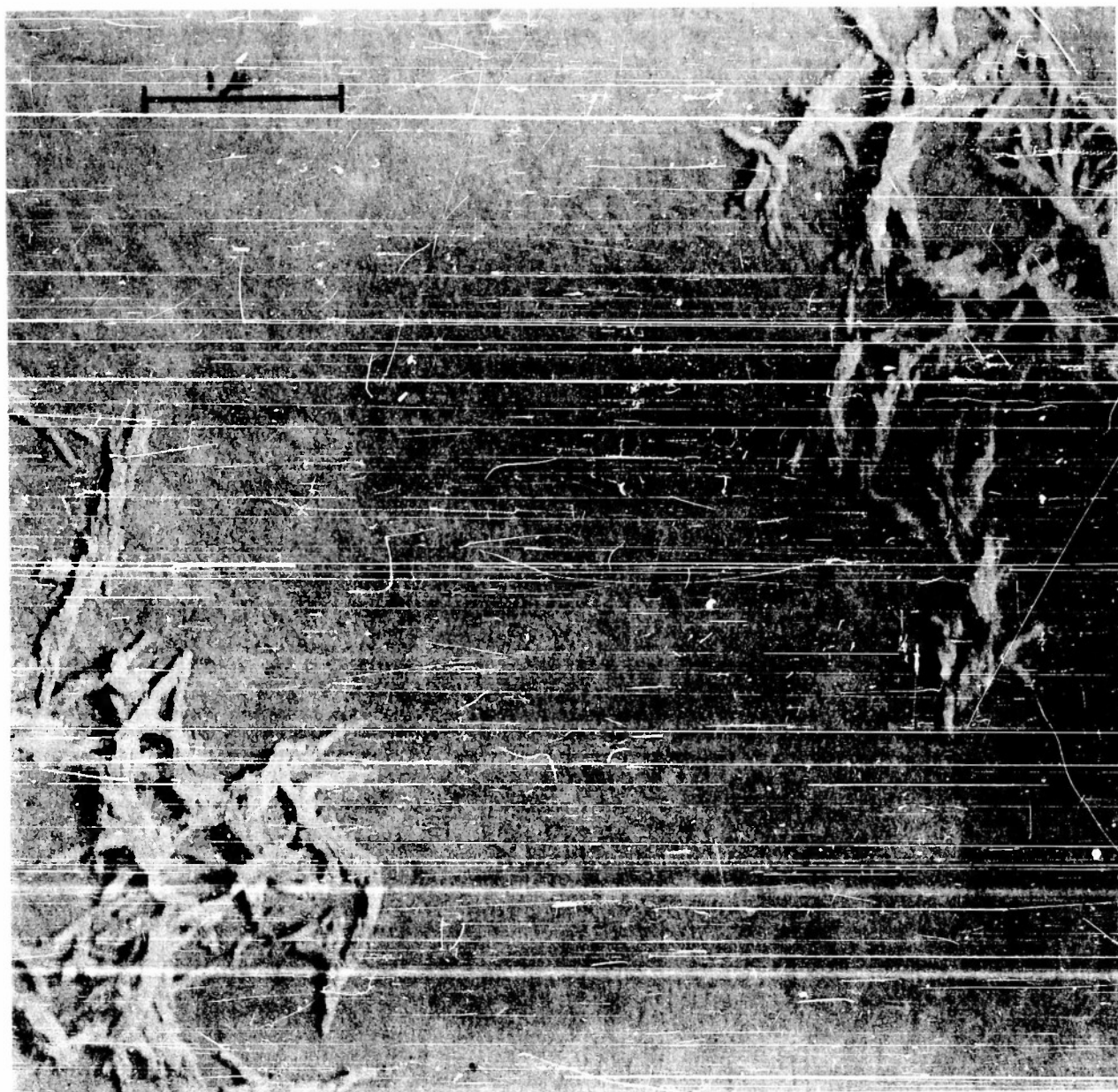
Magnification: 32,100 times
Scale: 1.14 in. = 1μ

Figure 1. Unworked Sample of MLC-6833 (12% Lithium 12 Hydroxystearate and 88% Tri decyl azelate)



Magnification: 32,100 times
Scale: 1.14 in. = 1 μ

Figure 2. Worked Sample (500 Strokes by Hain Microworker) of MLG-6833
(12% Lithium 12 Hydroxystearate and 88% Tri decyl azelate)



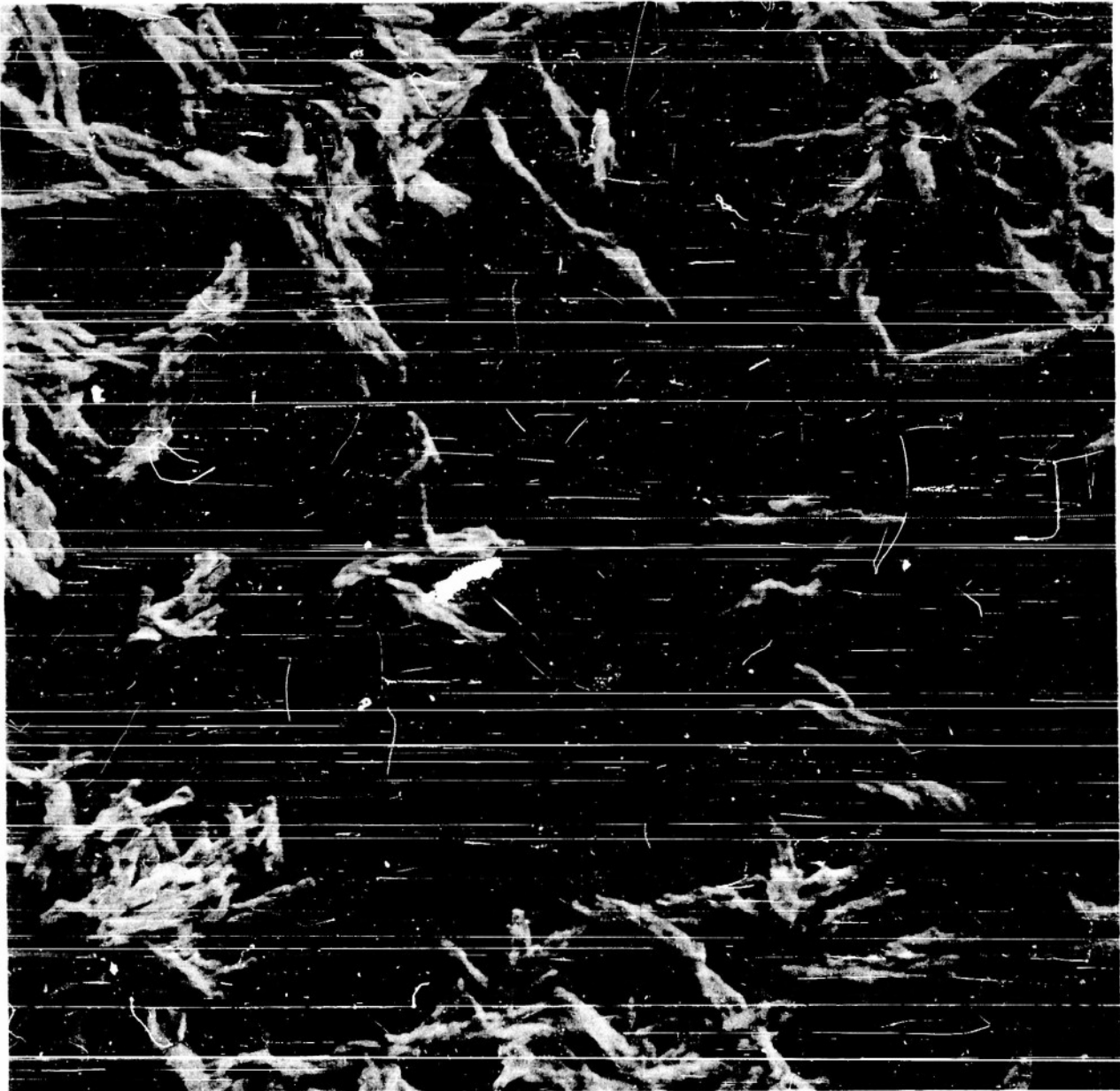
Magnification: 32,100 times
Scale: 1.14 in. = 1μ

Figure 3. Unworked Sample of MLC-6834 (12% Lithium 12 Hydroxystearate and 88% Dipropylene glycol dipelargonate)



Magnification: 32,100 times
Scale: 1.14 in. = 1μ

Figure 4. Worked Sample (500 Strokes by Hain Microworker) of MLG-6834
(12% Lithium 12 Hydroxystearate and 88% Dipropylene glycol dipelargonate)



Magnification: 29,000 times
Scale: 1.11 in. = 1μ

Figure 5. Unworked Sample of MLG-6812 (12% Lithium 12 Hydroxystearate and 88% Di (2 ethyl hexyl) sebacate)



Magnification: 32,100 times
Scale: 1.14 in. = 1μ

Figure 6. Unworked Sample of MLG-6836 (12% Lithium 9/10 Hydroxystearate and 88% Di (2 ethyl hexyl) sebacate)



Magnification: 32,100 times
Scale: 1.14 in. = 1μ

Figure 7. Worked Sample (500 Strokes by Hain Microworker) of MLG-6836
(12% Lithium 9/10 Hydroxystearate and 88% Di (2 ethyl hexyl)
sebacate)



Magnification: 32,100 times
Scale: 1.14 in. = 1μ

Figure 8. Unworked Sample of HLG-6838 (12% Lithium 9/10 Hydroxystearate and 88% DI (2 ethyl hexyl) sebacate)



Magnification: 32,100 times
Scale: 1.14 in. = 1μ

Figure 9. Worked Sample (500 Strokes by Hain Microworker) of MLG-6838
(12% Lithium 9/10 Hydroxystearate and 88% Di (2 ethyl hexyl)
sebacate)

Oil Separation

The greases were tested for oil separation in accordance with Federal Specification VV-L-791, method 35.1. Table 3 gives the different greases tested and the percentage by weight of oil lost.

TABLE III
OIL SEPARATION OF GREASES

IDENT NO.	WEIGHT LOSS (PERCENT)
MLG-6701	0.04
MLG-6702	5.72
MLG-6836	8.40
MLG-6703	5.34
MLG-6838	6.00
MLG-6704	2.05
MLG-6837	6.00
MLG-6753	5.40
MLG-6778	3.02
MLG-6705	5.17
MLG-6835	8.10
MLG-6754	5.30
MLG-6773	3.25
MLG-6714	0.06
MLG-6834	0.00
MLG-6755	1.10
MLG-6833	0.20

The greases made from lithium 12 hydroxystearate soap exhibited very small bleeding losses. The oil separation in lithium 9/10 hydroxystearate greases was much greater, and in several cases exceeded the maximum weight loss of 5% allowed under Military Specification MIL-G-3278, Grease; Aircraft and Instrument.

The large amount of oil separation in the lithium 9/10 hydroxystearate greases can be attributed to the percentage of soap present. Increasing the amount of soap

to produce a grease of a consistency comparable to that of a lithium 12 hydroxystearate grease would undoubtedly reduce the amount of oil separation.

Water Resistance

The lithium 12 hydroxystearate and the lithium 9/10 hydroxystearate greases were tested for their water resistance in the manner given by the following specifications:

Water Immersion Test, Military Specification MIL-G-3278, Grease, Aircraft and Instrument, and Water Resistance, Federal Specification VV-L-791e, Method 3252.1.

On the basis of the conducted tests, greases made with lithium 12 hydroxystearate have better water resistance than those made from lithium 9/10 hydroxystearate. Results of these tests are found in Table IV.

TABLE IV
WATER RESISTANCE OF GREASES

IDENT NO.	WATER RESISTANCE TEST (VV-L-791e, Method 3252.1) WEIGHT LOSS IN PERCENT	WATER IMMERSION TEST (MIL-G-3278, Par. 4.2.2.4.)
MLG-6701	5.97	Passed
MLG-6836	24.22	Passed
MLG-6838	15.73	Passed
MLG-6704	2.57	Passed
MLG-6837	27.98	Passed
MLG-6835	13.75	Passed
MLG-6834	1.98	Passed
MLG-6833	2.92	Passed

Wear Characteristics of Greases

The lithium hydroxystearate greases were tested on the Navy Gear Wear Tester in accordance with Military Specification MIL-G-3278, Grease, Aircraft and Instrument, Paragraph 4.2.2.8. Results of these tests are found in Table V.

TABLE V
WEARING CHARACTERISTICS OF GREASES

IDENT NO.	WEIGHT LOSS IN MILLIGRAMS (After 1000 Cycles)	
	5-Pound Load	10-Pound Load
MLG-6701	0.63	1.27
MLG-6836	0.54	1.15
MLG-6838	0.48	0.88
MLG-6704	0.46	1.40
MLG-6837	0.52	0.65
MLG-6705	0.59	1.20
MLG-6835	0.46	0.66
MLG-6834	0.55	1.22
MLG-6833	0.42	1.40

The wear characteristics of both types of the hydroxystearate greases are good on the basis of this test.

Oxidation Stability

Samples of the uninhibited greases were examined for their resistance to oxidation to determine the extent of oxidation of both the lithium 9/10 hydroxystearate and the lithium 12 hydroxystearate greases. The oxidation stability of the greases was determined by the Oxygen-Bomb method in accordance with Federal Specification WV-L-791, Method 345.3. Table VI indicates the results of this test.

TABLE VI
OXIDATION STABILITY OF GREASES

IDENT NO.	PRESSURE DROP
MLG-6833	74 psi in 100 hrs
MLG-6773	80 psi in 100 hrs
MLG-6778	80 psi in 100 hrs
MLG-6834	78 psi in 71 hrs
MLG-6835	77 psi in 88 hrs
MLG-6837	83 psi in 100 hrs

There was no appreciable difference in the rate of oxidation of any of the lithium hydroxystearate greases.

Copper Corrosion

All of the greases were tested for their resistance to copper corrosion, in accordance with Federal Specification VV-L-791, Method 530.9. All the lithium 12 and the lithium 9/10 hydroxystearate greases passed this test.

Melting Points

The melting points of the greases were determined, and are recorded in Table VII.

TABLE VII
MELTING POINTS OF GREASES

IDENT NO.	DROPPING POINT °F
MLG-6701	365
MLG-6702	352
MLG-6836	354
MLG-6703	367
MLG-6838	370
MLG-6704	372
MLG-6837	374
MLG-6753	374
MLG-6778	367
MLG-6705	365
MLG-6835	364
MLG-6754	365
MLG-6773	360
MLG-6714	369
MLG-6834	364
MLG-6755	372
MLG-6833	364

SECTION V

IMPROVEMENT OF MECHANICAL STABILITY

Several attempts have been made to improve the mechanical stability of the lithium 9/10 hydroxystearate greases. Three methods were used in an effort to improve their mechanical stability: (1) attempt to change the fiber-structure; (2) increase the amount of thickener; (3) add small amounts of other thickeners along with the lithium 9/10 hydroxystearate in preparing the greases.

Method #1

A grease was prepared using the following formulation:

15% lithium 9/10 hydroxystearate
85% di (2 ethyl-hexyl) sebacate

The soap and oil were added to a two neck flask fitted with a glass stirring rod and a thermometer. The flask was heated with a glascol heater controlled by a variac. This mixture was heated to the melting point (202°C) and held for 5 minutes. The solution was then chilled by pouring it onto a stainless steel table top and the grease milled on a three-roll roller mill with .002-in. clearance. This material was given the designation of MLG-9750. The mechanical stability of this grease was comparable to that of previous lithium 9/10 hydroxystearate greases.

Method #2

A soap was prepared by reacting 20% 12 hydroxystearic acid and 80% 9/10 hydroxystearic acid with the proper amount of lithium hydroxide. The soap which resulted was used in the preparation of a grease with the following formulation:

15% { lithium 12 hydroxystearate (20%)
 { lithium 9/10 hydroxystearate (80%)
85% di (2 ethyl-hexyl) sebacate

This grease was prepared in the same manner as MLG-9750. However, a melt was obtained at 180°C. The mixture was heated to 185°C and held for 5 minutes. The grease was assigned MLG-9801. The mechanical stability of this grease was better than MLG-9750, but it was not as good as that of the lithium 12 hydroxystearate greases.

Method #3

The grease formulation used in studying the effects of temperature on fiber-structure was the same as that of MLG-9750. Preparation of the grease was similar to that of MLG-9750. However, the mixture was heated well beyond the melting point (202°C) to 228°C and held for 5 minutes.

Mechanical stability of this grease, MLG-9771, was better than that of MLG-9801 and MLG-9750, and was comparable to that of lithium 12 hydroxystearate greases. A one pound batch of MLG-9771 was made using the same technique employed previously, and was given the identification number MLG-9918. The mechanical stability of this grease was tested as follows:

Unworked Penetration - ASTM Method

Worked Penetration - Federal Spec. VV-L-791, Method 31.1

Worked Stability - Federal Spec. VV-L-791, Method 31.3

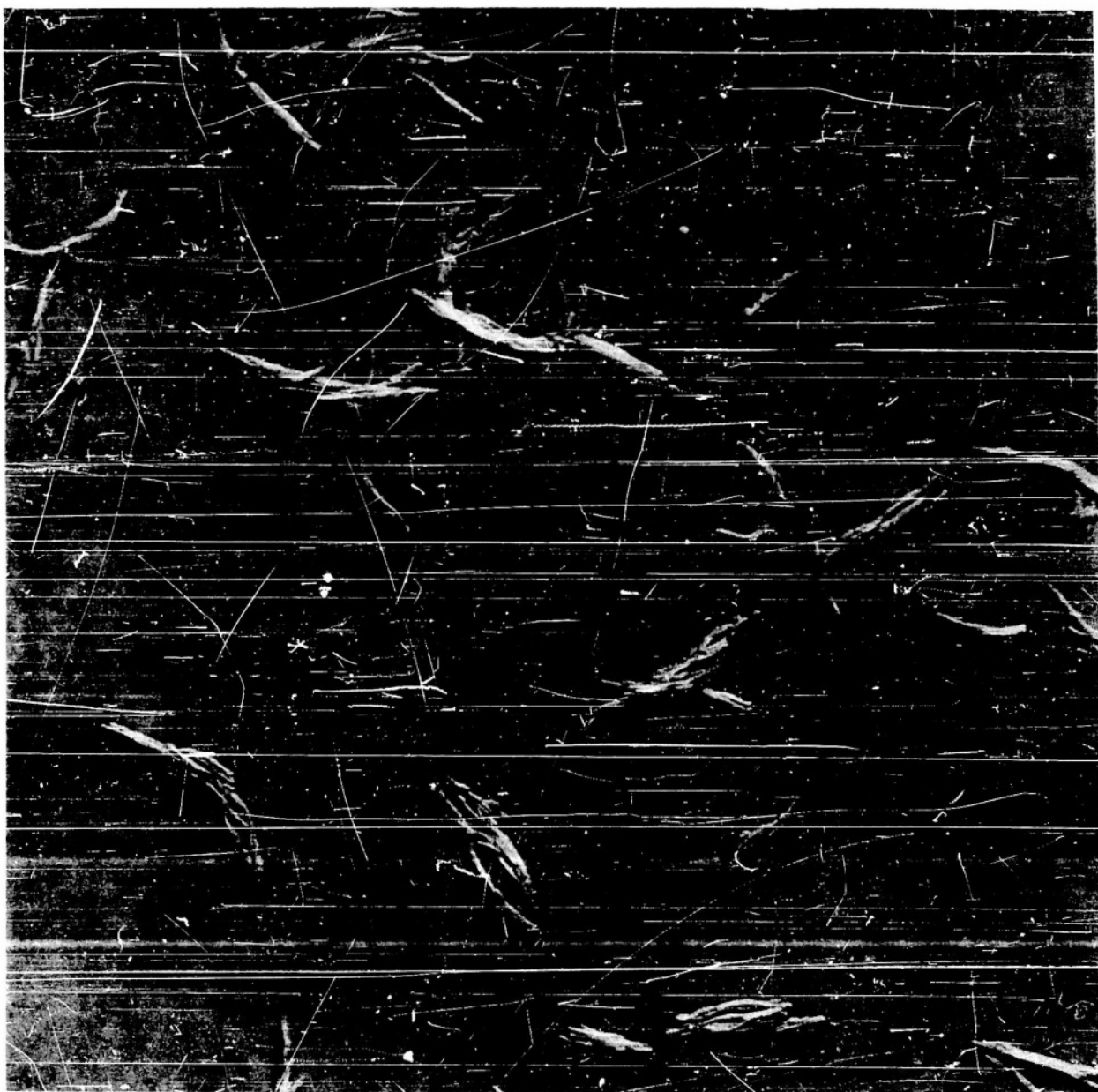
Data on the mechanical stability of the preceding greases are listed in Table VIII.

TABLE VIII
MECHANICAL STABILITY OF GREASES

MLG NUMBER	INITIAL ASTM MICROPENETRATION	STROKES WORKED IN MICROWORKER		UNWORKED ASTM PENETRATION	WORKED PENETRATION	WORKED STABILITY
		100	500			
9918	-	-	-	246 mm/10	260 mm/10	340 mm/10
9771	51	65	106	-	-	-
6701	62	71	94	-	-	-
9750	140	-	330	-	-	-
9801	59	90	130	-	-	-

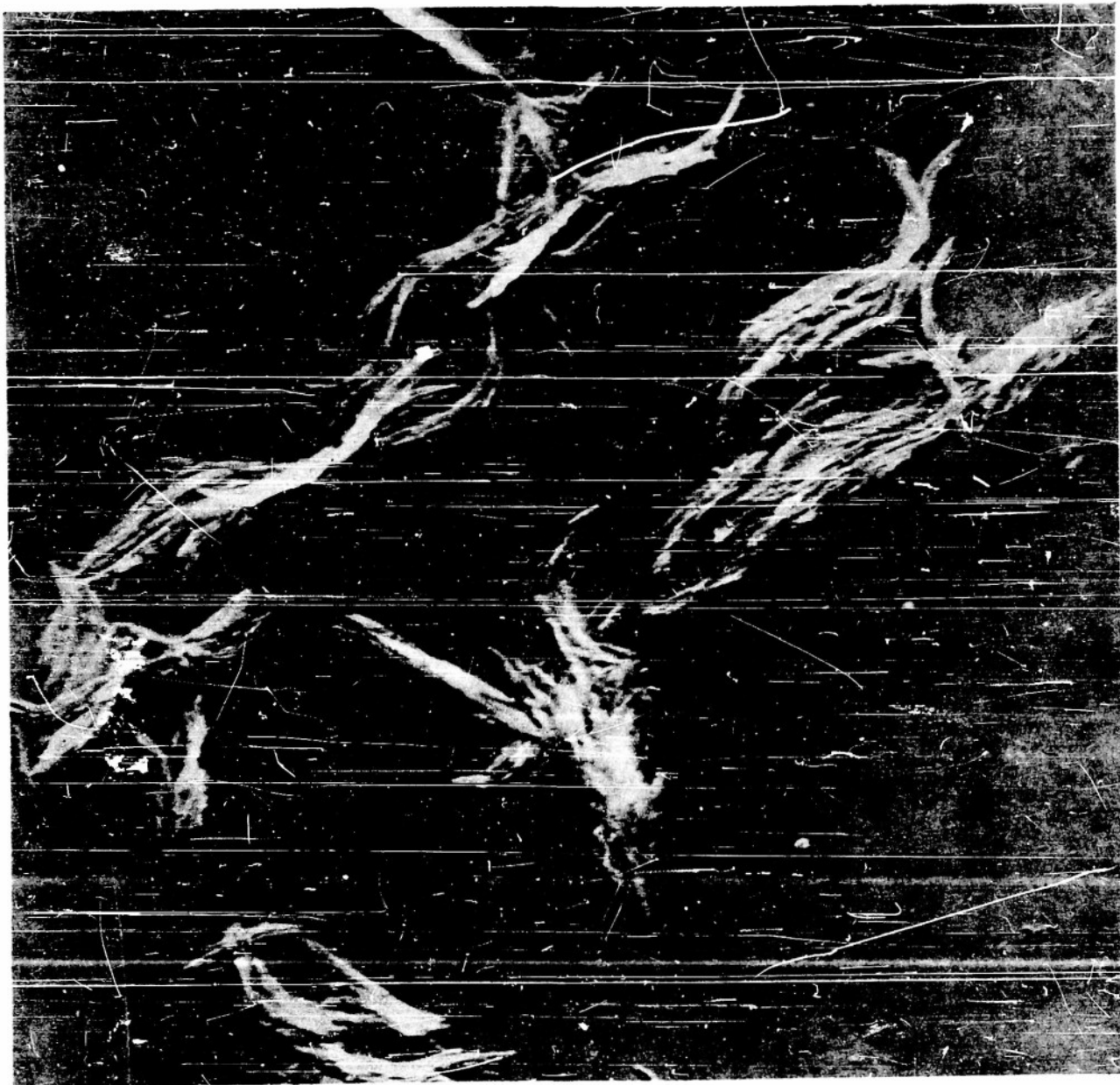
Electron micrographs were taken of MLG-9801 and MLG-9771. The unworked sample of MLG-9771 (Figure 10) revealed the same type of rod-like fiber previously encountered for lithium 9/10 hydroxystearate greases. However, the rods were not packed together in such large bundles and were well defined. The worked sample of MLG-9771 (Figure 11) shows the usual clumping of soap-fibers as a result of working the grease.

The fiber-structure of MLG-9801 (Figure 12) was similar to that of the other lithium 9/10 hydroxystearate greases. However, the fibers were better defined and the length to width ratio seemed to be increased.



Magnification: 29,000 times
Scale: 1.14 in. = 1μ

Figure 10. Unworked Sample of MLC-9771 (15% Lithium 9/10 Hydroxystearate and 85% Di (2 ethyl hexyl) sebacate)



Magnification: 29,000 times
Scale: 1.14 in. = 1μ

Figure 11. Worked Sample (500 Strokes by Hain Microworker) of MLG-9771
(15% Lithium 9/10 Hydroxystearate and 85% Di (2 ethyl hexyl)
sebacate)



Magnification: 29,000 times
Scale: 1.14 in. = 1μ

Figure 12. Unworked Sample of MLC-9801 (3% Lithium 12 Hydroxystearate, 12% Lithium 9/10 Hydroxystearate, and 85% Di (2 ethyl hexyl) sebacate)

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45579

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